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NATURAL DYES FROM LEAVES OF BUTEA MONOSPERMA AND ITS APPLICATION ON COTTON FABRIC, USING CHEMICAL AND NATURAL MORDANTS

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Original Article

ABSTRACT

In recent years, a growing interest in the revival of eco-friendly natural dyes has been manifested, due to environmental concern, as well as carcinogenic effects of synthetic dyes. Present investigation deals with the extraction of natural dyes from leaves of Butea monosperma and its application, on cotton fabrics using different concentrations (0.1 to 0.5%) of chemical and natural mordants. Chemical mordants used in the study were potassium aluminum sulphate, stannous chloride, ferrous sulphate, potassium dichromate, tannic acid and natural mordants, were almond shell, walnut shell, rinds of bahera fruits, rind of harad fruits, combination of almond shell + rinds of bahera fruits and combination of walnut shell + rinds of harad fruits. Dyeing was carried out by the pre - mordanting method. Large range of colour shades was obtained, because of varying mordant concentrations. Best dyed samples were selected on the basis of maximum colour strength, and were evaluated for their colour fastness properties viz. washing, rubbing, light and perspiration, showing fair to excellent fastness numerical ratings. The results showed the leaves as a good source of dyes.

KEYWORDS: Butea Monosperma, Leaves, Natural Dyes, Chemical and Natural Mordants, Cotton, Dyeing & Fastness Properties

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INTRODUCTION

Nature is full of fascinating colors and men had been exploiting them, for dyeing the garments, using them in food and many other items of the daily use. Colors in the day to day life of man are associated in the form of colored dress materials, hair, furnishings, upholstery etc. As far as natural colors and dyes are concerned, India has a very rich tradition of using natural dyes and also virtual monopoly, in their production and applications. Dyeing & printing were a craft, until mid of the nineteenth century. Numerous specimens of dyeing and printing of the ancient period are still available in the museums. The use of natural dyes/ colors has decreased due to the advent of synthetic dyes, whose consumption is esteemed around 1 million tons per annum, of which more than 50% were azo dyes (Stolz, 2001). It is only during the last two decades, when concern for the environment as well as carcinogenicity in synthetic dyes created an interest in biodegradable, eco-friendly, non-toxic and aesthetically appealing natural dyes, their production and R & D (Sewekow, 1988, Eom *et al*., 2001, Padhy and Rathi, 1990, Garg*et al*., 1991). Now, the whole world is looking back, towards the resurgence of natural dyes. Herbal dyes being naturally tended to be softer and their range of tones are very pleasant. At present, the total market of herbal dyes is to the tune of US $ 1 billion and is growing tremendously at the rate of 12%, per annum. Per capita

consumption of dyes is 400 g to 15 kg in developed and underdeveloped countries, for their utility in paints, inks, textiles, polymers, etc. India is a major exporter of herbal dyes mostly due to the ban on production of some of the synthetic dyes and intermediates in the developed countries, due to the pollution (Gaur, 2008 and Grover & Patni, 2011).

Butea monosperma (Lam.) Kuntze (Family: Fabaceae), is commonly known as *'Palash'* or *'Tessu'* or *'flame of the forest'* and various other names, in different parts of India and abundantly available in the forests of central India. It is one of the most beautiful trees and extensively used in Ayurveda, Unani and Homeopathic medicine and has become a cynosure of modern medicine. Plants of this genus are well known for their coloring (Sindhia and Bairwa, 2010). The flowers of the plant are well known for its yellow or orange natural dye. Since, the leaves of this plant are abundantly available and most part of it goes, it was planned to evaluate these leaves scientifically for their potential, as a natural dye. Therefore, the objectives of the study were to extract the natural dyes from the leaves of *Butea monosperma* and its application on cotton fabrics, using different types of chemical and natural mordants, for development of various types of color shades, which were further planned to be evaluated for their color fastness properties.

EXPERIMENTAL DETAILS, MATERIALS AND METHODS

Materials

The leaves of *Butea monosperma* were collected from nearby forest & village areas of Jabalpur district of Madhya Pradesh. The chemical mordants used in the study were of AR grade. Some natural mordants *viz.* Almond shell and Walnut shell, arranged from the local market and Bahera and Harad fruits, arranged from the forest areas were also in the study. Codes were assigned to all these mordants, given in Table 1. 100% pure mill white cotton fabric was used in the study. UV visible spectrophotometer was used for measuring the optical density. Distilled water was used for extraction and preparation of all chemical solutions and dyeing fabrics.

Methods

Preparation of Samples for Extraction of Dye

The leaves were dried in shade and grinded to get the powder form. The dried powder of the leaves was stored in polythene bags at room temperature (Bhuyan and Saikia, 2005, Hajare, *et al.*, 2013 and Rana *et al.*,2012).

Preparation of Natural Mordants

10g of each natural mordant powder was taken in 100ml distilled water (10% solution) and heated at 90^{o}C, by continuously stirring it for 1 hour, by using hot plate with magnetic stirrer. The volume of distilled water was maintained during heating. The resulting solution is filtered and concentrated to make extracts. These extracts were used for mordanting (Kateshkaran, 2015).

Pre-Treatment of Cotton Fabric

Pre-treatment of cotton fabric was carried out in 10% NaOH solution for 15 minutes, as per the method described by Grover and Patni, 2011.

Process Optimization for Extraction of Dye

Different amounts (2 - 14 g) of powdered leaves were placed in beakers containing 100ml of distilled water and each was boiled for 60 minutes over a gentle flame, maintaining a temperature of 90 – 95^{o}C, filtered and made with 100

ml. 1ml of this liquor was diluted to 100 ml again and the optimum amount of powdered leaves to water ratio, for the extraction of the dye was determined by measuring the optical density. Similarly, the time for extraction of dye from the leaves was also optimized, by optical density measurement (Dayal *et al.,* 2006 and Saxena *et al.,* 2012).

Extraction of Dye from Leaves

Based on the above experiment, the dye was extracted by heating the leaf's powder with distilled water, by maintaining a material to liquor ratio of 1:10 at 90-95°C for 60 minutes. The dye solution was filtered and evaporated to get the concentrated form. It was further dried in the sun, to obtain the solid dye.

Optimization of Different Parameters for Dyeing Cotton Fabric

Optimization of different parameters were carried out, following the methods described by Dayal *et al.,* 2006 and Saxena *et al*., 2012, with some modifications.

Determination of Optimum Dye Concentration for Dyeing Fabric

0.2, 0.4, 0.6, 0.8, 1.0, 1.2 and 1.4 of dye were taken in seven beakers and dissolved in 100 ml of distilled water. 1 ml of each solution was made up to 100 ml and an optical density was recorded. Seven fabric pieces were placed in seven beakers containing dye solutions, which were boiled for 60 minutes over a gentle flame, maintaining a temperature of 90-95^{o}C. The fabrics were taken out and liquors were filtered and made up to 100ml. 1ml of this liquor was diluted to 100ml again and the optical density was recorded for determination of optimum dye concentration, for dyeing the fabric.

Determination of Optimum Time for Dyeing Fabric

Cotton fabrics were placed in six beakers each, having 1% dye solution, and dye solutions were boiled for 10, 20, 30, 40, 50 and 60 mins, at 90 - 95^{o}C. Time of dyeing the fabric samples was determined on the basis of the depth of the colour, evenness of dye and brightness of the shade.

Determination of Optimum Weight of Fabric Sample

1.0, 1.25, 1.50, 1.75 and 2.0g of mordanted cotton samples were placed in 1% dye solution separately in five beakers and the solution was boiled for 30minutes, upto 90-95^{o}C. The quantity of fabric samples to be taken for dyeing was determined, on the basis of the depth of the colour, evenness of dye and brightness of shade.

Determination of Optimum Time for Mordanting the Fabric

0.2% solution of each mordant was prepared in distilled water and used for optimization of mordanting time. The scoured fabric samples were directly immersed in the mordant bath. The bath was heated to a temperature of 90-95°C for 10, 20, 30, 40, 50 and 60 mins, respectively. The bath was allowed to cool and the fabric was directly transferred to 1% dye bath, which was heated for 30 mins at 90-95°C for developing the colour. Time for mordanting the fabric samples was determined on the basis of the depth of the colour, evenness of dye and brightness of shade.

Mordanting

Pre-mordanting method was used for mordanting the cotton fabric. Five concentrations of 0.1 to 0.5% of each mordant viz. A1, A2, A3, A4, A5, A6, A7, A8, A9, A10 and A11 were prepared by dissolving 0.1, 0.2, 0.3, 0.4 and 0.5g of each mordant in 100 ml in distilled water separately and were used for mordanting. Five pieces of 1.25g of scouring cotton

fabrics were directly immersed in five mordant baths separately, which were heated to a temperature of 90 - 95°C for 30 mins. The baths were allowed to cool and the fabrics were directly transferred to dye bath, for developing the color shades.

Dyeing of Cotton Fabric with Flowers, Leaves and Bark Dyes

Method of mordanting and dyeing, as described by Nation *et al.*, 1977 was followed. Scoured cotton pieces, each having a weight of 1.25g were soaked in cold water for 30 mins to make even penetration of mordants. Desired concentrations of each mordant (0.1 to 0.5%) were dissolved in small amount of warm water and then transferred to a cold mordant bath. The pre-soaked cotton samples were put in it and allowed to simmer for optimum time, at 90 - 95°C temperature, over a gentle flame. After 20 mins, the bath was allowed to cool at room temperature. Mordanted samples were lifted out and were put into the 1% dye bath. The temperature of dye bath was raised upto 90 - 95°C, over a gentle flame. It was allowed to simmer for 30 mins, the dye bath was cooled to room temperature and the dyed cotton samples were taken out and washed in tap water and dried in shade. Scoured cotton fabric was also dyed without using mordant and was set as a control, to see the effect and to make the comparison with other dyed fabrics, using mordant.

Measurement of Colour Strength

The colour strength of the dyed cotton fabrics was determined by K/S values. The spectral reflectance of dyed samples was measured, using a CIE-Lab 1976 Illumination D65 (Observer 10 degrees). The K/S values were calculated by Kubelka-Munk equation.

$$\mathbf{K / S = (1 - R)^{2} / 2R}$$

Where, R is the decimal fraction of the reflectance of the dyed samples at λ_{max}. K is the absorption coefficient and S is scattering coefficient (Habibzadeh, 2010).

Selection of Colour Shades for Colour Fastness Properties

One colour shade out of five, developed using five concentrations of each mordant was selected on the basis of K/S value measurement. The selected colour shades were evaluated for colour fastness properties.

Determination of Colour Fastness Properties of Dyed Cotton Fabrics

The dyed samples were tested for colour fastness to washing, light, rubbing and perspiration, according to standard methods in the laboratory of Northern India Textile Research Association (NITRA, Ministry of Textiles, Government of India), Ghaziabad (UP). The colour fastness to washing was determined according to IS/ISO: 105 C10: 2006 A (1); light according to IS: 2454:1985 (Reaffirmed 2010); rubbing according to IS: 766: 1988 (Reaffirmed 2009) and perspiration according to IS: 171: 1983 (Reaffirmed 2009). Color change of fabrics was assessed against grey scale rating (ISO 104 A03: 1993). All the tests have been carried out in standard atmospheric condition of R. H. 65%±2% and temperature 27˚C±1˚C.

OBSERVATIONS, RESULT AND DISCUSSIONS

It is evident from the Table 2 and 3 that, 10g of dried leaves powder of *Butea monosperma* should be boiled in 100 ml of distilled water (material liquor ratio 1:10) at 90 -95°C for 60 mins, for maximum dye extraction. The yield of dye was 6.8%. The data in Table 4 and 5 showed that, 1% dye concentration and 30 mins time at 90 -95°C were the optimum conditions, for getting better color shade. The optimum weight of cotton sample was 1.25g for dyeing in 1% dye

solution. The visual observations also indicated that, for pre-mordanting, material to liquor ratio of 1:100 and 20 mins time was found to be optimum.

Development of Color Shades

A total of 56 color shades, 55 shades in combination with different concentrations of chemical and natural mordants and one shade without using mordant were developed. The Unmordant dyed sample was coded as WML and mordanted samples were coded as A1L, A2L, A3L, A4L, A5L, A6L, A7L, A8L, A9L, A10L and A11L, based on the codes of mordants (Fig. 1).

Measurement of Color Strength and Selection of Color Shades

Color strength (K/S values) of shades, developed using five different concentrations (0.1 - 0.5%) of each mordant and dye was given in Table 6. One color shade with maximum color strength value out of five shades, developed using each mordant was selected for measuring the color fastness properties. The names of selected color shades, along with their mordant concentrations are given in Table 7.

Colour Fastness Properties of Dyed Fabrics

According to the new system, the fastness ratings are described as (Booth, 2005):

- Very strong (Loss of dyes too much, Poor)
- Strong staining (Loss of dyes more, Fair)
- Marked staining (Loss of dyes visible, Good)
- Slight staining (Very little loss of dyes, Very Good)
- No staining (No loss of dyes, Excellent)

Numerical ratings for color fastness properties of unmordanted and mordanted dyed samples are given in Table 8.

Color Fastness to Washing

Fastness numerical rating for color staining of two adjacent fabrics, for all samples was 4-5, which showed very good to excellent fastness to washing. Fastness numerical rating for a change in color was 1 for unmordanted sample WML, which showed poor fastness to washing. Highest fastness numerical rating for a change in color was 4 for A5L sample, which corresponds to very good.

Color Fastness to Rubbing (Dry and Wet) of Dyed Fabrics

The result showed that, the fastness numerical ratings for all samples under dry conditions were 4-5, which showed very good to excellent fastness to rubbing, while the fastness varied from 2-5 under wet conditions, which corresponds to fair to excellent fastness.

Color Fastness to Light

One of the most important drawbacks of natural dyes is insufficient light fastness (Ismal and Yildirim, 2012). It was observed from results that, A3L and A5L samples exhibited the highest light fastness 4, which corresponds to very good. Light fastness values of all other samples range from 2-3, revealing fair to good fastness.

Color Fastness to Perspiration (Acidic & Alkaline)

It can be seen in Table 8 that, the fastness numerical rating for color staining of two adjacent fabrics for all samples was found to be 4-5, which indicated very good to excellent fastness to perspiration (acidic). A2L, A3L and A5L samples had the highest fastness numerical rating of 3-4 to change in color, which showed good to very good fastness.

The numerical ratings for color staining of two adjacent fabrics for all samples was found to be 4-5, which indicated very good to excellent fastness to perspiration (alkaline). It was observed that, for perspiration (alkaline), A2L, A3L and A5L samples were found to have the fastest numerical values 3-4, for changes in color similar to perspiration (acidic).

Measurement of Ratio of K/S Values

In order to find out the suitability of leaves dye extracts for dyeing, the unmordanted and mordanted dyed samples were compared to get the K/S values. The relative increase in K/S values, after pre-mordanting was expressed in terms of the ratio of K/S values of mordant dyed to un-mordant dyed cotton fabrics (Table 9). All mordanted samples except A6L (0.32) were found to contain higher K/S values, as compared to unmordanted sample, WML (0.35).

The relative increase in K/S expressed in terms of the ratio of mordanted to unmordanted samples, is also seen in the case of above discussed samples. From Table 9, it can also be concluded that, in case of unmordant dyed fabric having more K/S value, its corresponding dye extract had a strong affinity to cotton. Most of the metal salts exhibited the highest K/S values, due to their abilities to form co-ordination complexes, with dye molecules. This strong tendency of metal salts, enhanced the interaction between the fiber and the dye, resulting in high dye uptake (Jothi, 2008). The effects of mordants on color values of dyed fabrics are shown in Fig.3.

In the present investigation, the dye was extracted from leaves through the aqueous medium in an eco-friendly manner and all dyeing experiments have been made with natural fabric i.e. 100% pure cotton. Pre-treatment of cotton fabric with sodium hydroxide solution and washing in running water removed all extra staple fibers, and natural impurities like oils, fats, waxes and coloring matter, remnants of the added matter, starch and stains of dust or grease, if any (Grover and Patni, 2011). Proper washing of fabric facilitated the fixation of dye in a uniform manner. Numerous dyeing experiments have been performed in view to obtain various color shades, with good fixation properties. The development of different color shades using various mordants, is due to the formation of complexes between the polyphenols and organic compounds in the dye, with metal ions of mordants, which absorbs in different regions of the visible spectrum leading to different colors (Dayal *et al.*, 2006 and Saxena *et al.*, 2012). Pre-mordanting, dye bath concentration and dyeing duration are some of the important aspects, which enable uniform and smoothing color effects to the fabric. In present context of dyeing, natural mordants *viz.* myrabalon extracts (Baheda and Harad), Almond and Wallnut shell extracts and chemicals in safe limits *viz.* Potassium aluminium sulphate, ferrous sulphate, potassium dichromate, tannic acid and stannous chloride were used. No salts of heavy metals like Ni, Cr, Pb, Co, Mn etc. were used. Although, the use of heavy metal salts could provide unlimited fast shades, but there is no sense to use these in the context of environmental and health hazard considerations (Sachan and Kapoor, 2007).

Natural mordants were used in the study, as they are safe and eco-friendly. Besides, these are the good source of tannin and tannins have good fixing properties. In this study, chemical mordants in very low concentration (0.1 to 0.5%) were used, due to pollution and environmental concerns. Fastness numerical rating for washing, light, rubbing and

perspiration were found quite good to excellent, for most of the samples. However, A3L and A5L samples showed the best fastness ratings. Natural mordants were found equally effective, as compared to chemical mordants. All mordanted samples, except A6L had higher K/S values, showing strong affinity to cotton compared to control WML.

CONCLUSIONS

Present work showed that, the leaves of *Butea monosperma* can be used as dye, for colouring cotton fabrics which will help to augment the export of natural dyed garments to the countries, where the use of azo dyes has been banned. Since, the use of heavy metals in dyeing is discarded; this dye and dyed textiles will not cause any skin problems to the wearer and also will not pollute the environment. Leaves dye can also be utilized for dyeing handloom and craft products, prepared by craftswomen for enhancing their livelihood.

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Table 1: Chemical and Natural Mordants and their Codes

SI. No.	Name of Mordants	Codes
1	Potassium aluminium sulphate	A1
2	Ferrous sulphate	A2
3	Potassium dichromate	A3
4	Tannic acid	A4
5	Stannous chloride	A5
6	Almond shell	A6
7	Walnut shell	A7
8	Rinds of Bahera fruits	A8
9	Rinds of Harad fruits	A9
10	Mixture of Almond shell + rinds of Bahera fruits (50+50 ratio)	A10
11	Mixture of Walnut shell + rinds of Harad fruits(50+50 ratio)	A11

Table 2: Optimization of Leaves Powder Concentration

S. No.	Weight of Leaves Powder (g)/ 100 ml of Water	Extraction Time (minutes)	Wave Length (nm)	Optical Density
1	2	60	268	0.31
2	4	60	268	0.52
3	6	60	268	1.002
4	8	60	268	0.765
5	**10**	**60**	**268**	**1.121**
6	12	60	268	0.943
7	14	60	268	0.905

Table 3: Optimization of Time for Leaves Dye Extraction

S. No.	Weight of Leaves Powder (g)/ 100 ml of Water	Extraction Time (minutes)	Wave Length (nm)	Optical Density
1	10	15	268	0.346
2	10	30	268	0.546
3	10	45	268	0.576
4	10	60	268	0.798

5	10	75	268	0.782
6	10	90	268	0.781
7	10	105	268	0.711

Table 4: Percentage of Absorption in Different Dye Concentrations by Cotton Fabric

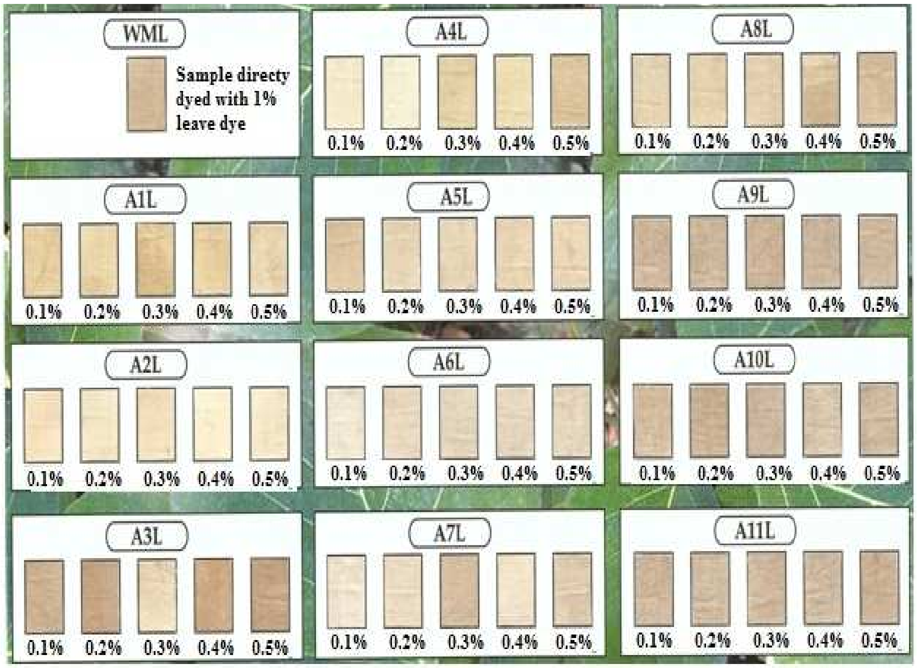
S. No	Wavelength (nm)	Time (min)	Dye Solution (%)	Wt. of Cotton (g)	OD before Dyeing	OD after Dyeing	Percentage of Absorption %
1	268	60	0.2	1.25	0.22	0.19	13.63
2	268	60	0.4	1.25	0.426	0.383	10.09
3	268	60	0.6	1.25	0.626	0.536	14.37
4	268	60	0.8	1.25	0.756	0.632	16.402
5	268	60	1	1.25	1.12	0.89	20.53
6	268	60	1.2	1.25	0.238	0.19	20.16
7	268	60	1.4	1.25	0.258	0.21	18.6

Table 5: Percentage of Absorption for Optimization of Time for Dyeing Fabric

S. No.	Wavelength (nm)	Time (min)	Dye Solution (%)	Wt. of Cotton (g)	OD before Dyeing	OD after Dyeing	Percentage of Absorption %
1	268	10	1	1.25	0.11	0.09	18.18
2	268	20	1	1.25	0.13	0.11	15.384
3	268	30	1	1.25	1.02	0.79	22.55
4	268	40	1	1.25	0.12	0.1	16.66
5	268	50	1	1.25	0.16	0.13	18.75
6	268	60	1	1.25	1	0.82	18

Table 6: Colour Strength of Leaves Dye on Cotton Fabric

S. No.	Mordant Concentration (%)	Colour Strength (K/S Value) of Samples										
		A1L	A2L	A3L	A4L	A5L	A6L	A7L	A8L	A9L	A10L	A11L
1	0.1	0.57	0.39	2.6	0.36	**1.11**	0.29	0.5	0.86	0.81	0.59	0.8
2	0.2	0.31	**0.42**	**2.17**	0.26	0.54	**0.32**	0.51	1.21	0.76	**0.86**	**0.81**
3	0.3	0.45	0.4	1.48	**0.37**	1.02	0.29	0.6	**1.22**	0.54	0.8	0.76
4	0.4	0.54	0.36	1.42	0.33	0.5	0.3	**0.68**	0.49	**0.92**	0.79	0.76
5	0.5	0.5	0.41	1.39	0.32	0.72	0.12	0.61	0.89	0.9	0.81	0.79



Note: 0.1% to 0.5% represents mordant's concentration

Figure 1: Colour Shades Developed on Cotton Fabrics from Leaves Dye

Table 7: Names of Selected Colour Shades

S. No.	Samples Code	Mordant Concentrations (%)	Name of Preferred Colour Shades
1	WML	-	Sand castle
2	A1L	0.1	Shortbread
3	A2L	0.2	Oat
4	A3L	0.2	Sand
5	A4L	0.3	Sand castel
6	A5L	0.1	Beige
7	A6L	0.2	Tan
8	A7L	0.4	Hazelwood
9	A8L	0.3	Tan
10	A9L	0.4	Dark Hazelwood
11	A10L	0.2	Dark Hazelwood
12	A11L	0.2	Dark Hazelwood

Table 8: Colour Fastness Properties of Dyed Cotton Fabrics

S. No.	Samples Code	Mordant Concentrations (%)	Wash Fastness		Rub Fastness		Light	Perspiration			
								Acidic		Alkaline	
			CC	CS	Dry	Wet		CC	CS	CC	CS
1	WML	-	1	4-5	4-5	4-5	3	1	4-5	2	4-5
2	A1L	0.1	1	4- 5	4- 5	3-4	3	1-2	4-5	3	4-5
3	A2L	0.2	1-2	4-5	4-5	4-5	2-3	3-4	4-5	3-4	4-5
4	A3L	0.2	2-3	4-5	4-5	2-3	4	3-4	4-5	3-4	4-5
5	A4L	0.3	1	4-5	4-5	4-5	2	1	4-5	1	4-5
6	A5L	0.1	4	4-5	4-5	3-4	4	3-4	4-5	3-4	4-5
7	A6L	0.2	1	4-5	4-5	4-5	2-3	1	4-5	1-2	4-5
8	A7L	0.4	1	4-5	4-5	3-4	2-3	2-3	4-5	2-4	4-5
9	A8L	0.3	1	4-5	4-5	4	2-3	2-3	4-5	2-3	4-5

Table 8: contd.,

10	A9L	0.4	1-2	4-5	4-5	4	3	2-3	4-5	2-3	4-5
11	A10L	0.2	1-2	4-5	4-5	3-4	3	2-3	4-5	3	4-5
12	A11L	0.2	1	4-5	4-5	3-4	3	2-3	4-5	2	4-5

CC = Change in Colour, CS = Colour Staining

Table 9: Colour Strength (K/S) Values of Dyed Cotton Fabrics

S. No.	Samples Code	Mordant Concentrations (%)	K/S Value	K/S Ratio
1	WML	-	0.35	
2	A1L	0.1	0.57	1.54
3	A2L	0.2	0.42	1.20
4	A3L	0.2	2.17	6.20
5	A4L	0.3	0.37	1.05
6	A5L	0.1	1.11	3.17
7	A6L	0.2	0.32	0.91
8	A7L	0.4	0.68	1.94
9	A8L	0.3	1.22	3.48
10	A9L	0.4	0.92	2.62
11	A10L	0.2	0.86	2.45
12	A11L	0.2	0.81	2.31

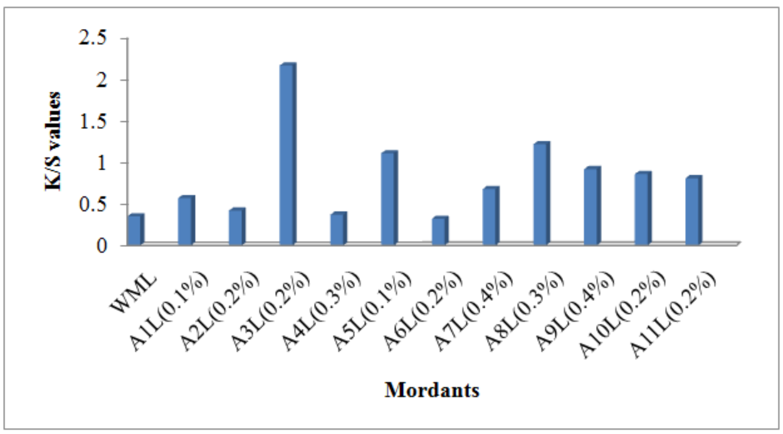


Figure 3: Effects of Mordants on Colour Values of Dyed Cotton Fabrics